FOCUSED PROJECT
NON-PROPRIETARY RESEARCH AGREEMENT
APPENDIX A –Melt Microfluidic Multi-sample Rheometer (M^3R)

Article 1. **INTRODUCTION**

Melt rheometry is essential to the Polymers Industry because it provides critical information regarding the processability of new materials, and provides insights into a wide-range of fundamental material properties. Combinatorial methods enable the synthesis of large numbers of new polymers and the fabrication of libraries of polymer blends. However, traditional rheometry techniques are not designed for the rapid measurement of a large number of samples. Moreover, traditional rheometry techniques are not compatible with the small samples produced by combinatorial techniques. In response to these needs, researchers at the NIST Combinatorial Methods Center have developed a conceptual design for a high throughput melt rheometer. Research through this Focus Project will develop our promising design into a robust high-throughput metrology that incorporates the following features:

- Simultaneous measurement of at least eight polymer melt samples.
- Sample size less than 25 mg.
- Amenable to automated sample loading.
- Temperature range that enables melt rheometry over most classes of polymers, with improved temperature control over traditional methods.
- Measurement of wall slip and entry flow effects.
- Low implementation barriers due to a) lack of moving mechanical parts, and b) cleanable/disposable device components.

Article2. **BACKGROUND**

Two principle classes of rheometers have wide-scale use in polymers research: capillary rheometers and rotational rheometers. Rotational rheometers, which are typically found in academic environments, offer the advantage of acquiring dynamic moduli over a range of frequencies, but has the disadvantages of time consuming sample preparation, difficulty of use, large sample size (approximately 500 mg), and of single sample capability. The second type, capillary rheometers, are more commonly used by industry because they offer simpler measurement protocols and operate at higher shear rates. The primary disadvantages are: large sample size (approximately 25 g), the fact that only one or two samples are measured at a time, and the difficulty of measurement at low shear rates. These two classes of rheometers are thus not amenable to the high throughput approach being developed in many laboratories. The development of a simple high-quality polymer melt rheometer which can simultaneously measure multiple samples of dramatically decreased mass would be of broad-based value to this community.

In our design, rheological measurements will require approximately 1000 times less material than traditional capillary rheometers and will be able to measure eight samples simultaneously. Termed the Melt Microfluidic Multi-sample Rheometer (M^3R), it is based on capillary rheometry but operates over a shear rate range more comparable to a rotational rheometer. It is designed to work on molten polymeric materials over a broad temperature range. The M^3R offers the simplicity of capillary rheometry and a design that enables parallel measurements demanded by a combinatorial and high-throughput laboratory.

A measurement of polymer rheology based on a capillary rheometer is quite simple in principle, but will involve a significant engineering effort to implement for high-throughput implementation. First one needs an accurate measure of the smallest dimension (the one that most actively constricts the flow.) In

traditional capillary rheometry, the size of the minimum dimension is approximately 1 mm. Next one needs to work in one of two modes: pressure driven or flow driven. In a pressure driven mode, the pressure is actively controlled and the resulting flow rate is measured. One applies a sequence of increasing pressures and measures the resulting flow (typically with a timer and a weighing scale.) More commonly, one applies a known flow rate (with a piston in an upstream reservoir) and measures the resulting pressure.

The basic principle of our microfluidic slit rheometer is shown below. The simplicity of the device is based on the following concepts:

I. The flow channel is constructed by cutting numerous slits (one for each sample) in commercial grade of shim stock. As approximately 30 shim stocks can be cut (via EDM) simultaneously, the price per shim stock will be such that it is disposable (perhaps $20 per shim stock). This disposable shimstock is sandwiched in between a piece of steel and an optical window.

II. The flow is pressure driven; thus there are no moving parts and since the sample size is small, the pressure regulation is much simpler than traditional capillary rheometry. By using gas (e.g. N_2) as the pressurizing agent, we can pressurize multiple samples simultaneously.

III. Integrated design using a single housing will yield better temperature control.

IV. Measurement of flow rate is performed optically by monitoring the front of the polymer flow field as it first enters the channel and displaces air. Once the length of the flow front is greater than approximately 20 times the height of the channel, accurate measurements can be made.

Article 3. **COLLABORATION AND DISSEMINATION**

A discussion meeting will be arranged within six weeks of the formal launch of the project to define model systems for study and to discuss. NCMC will facilitate dissemination and communication among members of the focus project. Written reports will be sent to the members at six-month intervals, with updates more frequently via conference calls at three month intervals for the duration of the project. In order to facilitate the collaboration, specifications for methods, instruments, programs, data analysis, and other aspects of this work will be available to members during the course of the project. A summary report will be provided within two months of the end of the project. The NCMC labs will be open to prearranged visits from member scientists interested in hands-on participation in method development and technology transfer.

As with base level membership in the NCMC, all of the research carried out in the Focused Project is non-proprietary and is intended for publication in the public domain. No proprietary information or materials will be solicited or accepted by NIST from member organizations. The scope of the work by NIST included in this focused project will be limited to milestones described in the final focused project agreement.

Article 4. **DURATION OF AGREEMENT**

The Project period is 1.5 years. The nominal Focus Project launch date is July 15th, 2005. NIST reserves the right to re-define formal project launch and termination dates and the project timeline (Article 5) based upon the actual dates of receipt of project fees from member as defined in Article 7.

Article 5. **PROJECT MILESTONES AND TIMELINE**

Project timeline is based upon a total project period of 1.5 years (6 Quarters) and project launch and termination dates as defined in Article 5.

Q1:

- Design prototype for a four sample M^3R.
- Construct a four sample M^3R and begin proof of principle tests using NIST-supplied polyolefin specimens.
- Definition and transfer of appropriate polymeric materials for study from members.

Q2:

- Complete proof of principle testing for four sample M^3R prototype.
- Begin second level measurements: shear thinning behavior, entry/exit pressure corrections, and slip corrections.
- Estimate parameter space of the instrument (functional shear rate range, and applied pressure range, material viscosity range, temperature range).

Q3:

- Design prototype for an eight sample M^3R.
- Begin development of data acquisition software.
- Define data interpretation protocols; in particular construct a curve of viscosity as a function of shear stress / shear rate.
- Presentation of second level measurements in a professional meeting

Q4:

- Construct prototype for an eight-sample M^3R.
- Conduct validation experiments using the eight-sample M^3R.

Q5:

- Complete demonstration experiments on member supplied model specimens.
- Complete development of data acquisition software.

Q6:

- Written device designs and method protocols supplied.
- Methods transfer to member and/or engineering firm proxy.
- Demonstration of final device including high temperature capability, ability to measure eight samples simultaneously, user friendly software that integrates all device capabilities. Presentation in professional meetings, preparation of archival publication.

Article 6. **FINANCIAL OBLIGATION**

The cost of the project research (C) is $95 K per year.
The project is expected to last 1.5 years. Cost will be prorated after the first year.

The final membership fee is determined by the number of institutions (N) who agree to join the project. That is, the yearly fee will be C/N, e.g. $31.6 / year, if three institutions join. Upon public announcement of the project, institutions will have 6 weeks to join the project. If only one institution joins the project, that institution is expected to pay the entire cost of the project, C, per year for the project to be launched.